

# **Cleaning to the Monolayer Level**

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## **Abstract**

Laser and instrument systems used for space flight have extreme requirements for cleanliness. Many of these systems specify or require cleanliness target values approaching one monolayer of non-volatile residue (NVR) or even less in some cases. This opens up a completely new series of challenges that are added to the challenges facing contamination control and contamination analysis personnel.

As the amount of molecular contamination on a surface approaches zero, the behavior of the contaminant changes. These behavior changes require knowledge of the surfaces and the contamination beyond whether bulk material is soluble in the solvent. As the thickness of the contamination drops below a few monolayers, the bulk properties become nearly irrelevant. Knowledge of the interactions of the contaminant with the surface becomes critical. This includes both the equilibrium and kinetics of the surface adsorption. The paper will address the fundamental physical, procedural, philosophical, and technical aspects of cleaning surfaces to the monolayer level.

## **Introduction**

In the development of the present state of the art in contamination control, the author has utilized the knowledge base of hundreds of years of science and technology as they relate to the interactions of molecules with materials. The starting point for this effort began with the historical work of Stowers and others<sup>1</sup>, and employed state of the art detection technology for the verification of cleanliness levels. In addition, it brought into play fundamental knowledge of surface chemistry, surface analysis and expertise of personnel working at the forefront of contamination control for spaceflight systems. All of this information was tempered by solid measurement of contamination levels, and identification of the contamination composition and its sources. The minimum quantifiable non-volatile residue levels were lowered by a factor of one million, to meet the requirements of this project.

This paper will attempt to provide the basis for the development of strategies and procedures for attaining extreme cleanliness levels with a minimum risk to components. It is impossible to provide generic cleaning procedures that will work in all cases.

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<sup>1</sup>I.F. Stowers and H.G. Patton, Lawrence Livermore Laboratory Preprint UCRL 80730, 1978

However, it is felt that these guidelines will provide invaluable information on the attainment of the desired cleanliness levels.

This work is based upon a number of years effort on space flight laser projects where the contamination degradation of the surfaces by the outgassing internal components was to be held at less than a monolayer at end of mission. In these cases, measured NVR deposition rates on a quartz crystal micro balance indicated less than 0.01 nanograms for ten hours when held at a temperature over 50 degrees centigrade lower than the laser temperature. This indicates that the outgassing of the laser cavity of a nominal 300 gram per mole contaminant would result in less than a monolayer over five years, even assuming worst case thermal cycling from lower survival limit to upper survival limit. This resulted in no detectable contamination on a cold plate to a sensitivity of 100 picograms per component from a 15 hour collection. Particulate levels in the laser cavity were maintained at levels less than 50 for the laser cavity through process control and mitigation of contamination sources. Better particulate levels could have been achieved if the hardware had not been already built.

## **Interactions of Matter**

The interaction of matter with other matter is controlled by the polarizabilities of the matter.<sup>2</sup> At the rudimentary level, the interaction of surface molecule A with contaminant molecule B is defined by the interaction of the electronic configuration of A with the electronic configuration of B, and the electronic configuration of B with the electronic configuration of A<sup>3</sup>. These configurations are controlled by the properties of the individual electronic configurations of the wavefunctions of the supermolecule of the surface segment, other adsorbed materials, the adsorbed contaminant, the energetics of the net environment and the arrangement of the molecules in space. As a result, the behavior of contaminant molecule B is no longer just a function of the configuration of B.

Typical surface interaction levels are significantly greater than the interactions of molecule B with like molecules. An easily measurable example of this behavior is the surface adsorption of water in an ultrahigh vacuum system. Water adsorbed on a surface within a vacuum system, the dew point of the water present at the monolayer level is raised many orders of magnitude due to the significantly greater attractive forces between the water and the surfaces in the system. The rate of loss of water from the surfaces, assuming a sufficiently large pumping speed, becomes only a function of the interactions of the surfaces, the thickness of the water adsorption layer, and the energetics of the system. A residual gas analyzer may be used to measure the partial pressure of water in the system. This shows the effective partial pressure of water in the system. Proving that the partial pressure of water at 300K will be decreased many orders of magnitude due to the lower energy state of the water adsorbed on the surface. Thus, the properties of an

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<sup>2</sup>L. R. Snyder, Principles of Adsorption Chromatography, Marcel Dekker, New York, 1968.

<sup>3</sup>A. J. Stone, The Theory of Intermolecular Forces, International Series of Monographs on Chemistry 32, Oxford University Press, Oxford, 1996.

adsorbed molecule will be functionally controlled by the surface conditions, not the behavior of bulk material.

Within a homologous series of chemical compounds, the polarizability of the molecules is a function of the molecular mass. Increases in the size and mass of a contaminant will increase the adhesion of the molecule on the surface. In addition, the polarizabilities as qualitatively described in hard acid/soft acid theory will affect the adsorption on the surface. In a nutshell, the larger the atomic radii within a molecule, the more that it will be distortable by an intermolecular interaction, and the more energetically favorable it will be to be adsorbed rather than free.

In the case of contaminants with multiple interaction sites, it becomes necessary for the interactions of all sites to be broken simultaneously.<sup>4</sup> This results in a higher order interaction of the contaminant with the surface. This interaction is exactly analogous to the interaction of a chelating agent with an ion. In these cases, the interaction is of the order of the number of strongly interacting sites. Thus, a contaminant with three interacting sites is not three times more difficult to remove than a homolog with one site, but rather it scales to the third power, assuming equal interaction levels of the three sites. This also leads to the introduction of a kinetic effect in the removal of the molecules from the surface.

## **Initial activities**

Before beginning any activity where contamination control is required, it is essential to open a dialog between the contamination control personnel; the cleaning personnel, the analytical testing lab personnel, quality control personnel and the project lead personnel. This is required to assure that all of the involved personnel know what the requirements are, gain their buy-in and assure that they are able to provide support to ensure that this level can be met. If there are issues with the requirements with these personnel, find someone else who is able to provide the support to the necessary level. At this point, the definition of the cleaning procedures can begin.

It is not possible to define one cleaning process that will be suitable or adequate for all components and contaminants.

Never assume anything. The piece is loaded with contaminants until you prove otherwise.

## **Modeling the interaction**

In evaluating the strength of interactions of contaminant molecules with surfaces, there are a number of options. The best model being the system itself. In most cases, repeated cleaning of an optic or subassembly imparts too high of a risk to be feasible. If witness

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<sup>4</sup>H.L. Laitinen and Harris, Chemical Analysis, McGraw-Hill, 1975.

samples, rejected optics or exemplar pieces are available, they provide an extremely valuable option for testing contamination abatement options. In these cases, the presence of defects in coatings or substrates will provide enhanced adhesion of contaminants. The enhanced adhesion of the contaminants and potentially decreased coating stability will provide an additional level of certainty to the efficacy of the cleaning and its effects upon the optic. It should be noted that if additional damage appears in a defective optic, that the damage may have been latent damage, (existing but not detectable,) and not due to the cleaning process. This has been seen with the cleaning of old optics where subsurface damage has propagated resulting in tightly held optic fragments being removed. It should be noted that it is better to remove them with cleaning rather than with the laser, risking propagation of damage through the laser optic train.

If degradation of optical quality is seen, due to a cleaning process, the optic and cleaning process should be analyzed by a chemist or materials engineer familiar with glass and optical coatings chemistry. If there is no apparent reason for the degradation of the coating/surface, a pristine example of the optical combination from the same vendor should be tested. A well-maintained coating witness sample makes a suitable test piece. In the case of a non-optic surface, a chemist or materials engineer should be likewise be consulted.

If no visible degradation of the component has occurred, the test articles should be evaluated for cleanliness through adequate measurement techniques to assure the level of cleanliness. This will involve quantitative measurements of surface particulates, either in situ via microscopic counting in a clean bench or via tape lift. (Verification has been made that in the case of clean dry optics, tape lifts followed by thorough cleaning of the surface with spectroscopic grade isopropyl alcohol does not leave any detectable NVR on the optic.) Measurement of the NVR either by direct surface measurement or by repeated sampling with an appropriate solvent followed by GC/MS with an appropriate method, will allow the determination of the state of cleanliness.

In the determination of the adequacy of a solvent to remove the existing contamination from the surface of a part it is necessary to have a “measuring stick” for the efficacy of a solvent. The utilization of normal phase chromatography, thin layer chromatography, or multiple sampling or direct surface analysis of a clear anodized aluminum surface will provide a model for a strongly interacting surface. These cases provide the strongest normally available surface adsorption media. They are also readily relatable to most surfaces. In addition they are highly reproducible, allowing the relation of behavior of different contaminants with different solvents.

Knowledge of adsorption chromatography can be valuable in the selection of conditions for removing contaminants from surfaces. This is based upon the theoretical and practical aspects of chromatography<sup>1</sup>. The interactions of molecules with the solid adsorption matrices provides a direct relation to the behavior of the trace and ultra trace amounts of non-volatile residues on the surface oxides and polar materials in a directly relatable manner, based upon oxide thickness and activity. A great deal of effort was expended in

describing the behavior of molecules in these systems, which is often lost as adsorption based supports fell out of favor. Nevertheless, reading L.R. Snyder's Adsorption Chromatography would be well worth while<sup>1</sup>.

From the practice of normal phase adsorption chromatography, it has been learned that the combination of a highly polarizable low polarity solvent and a miscible polar solvent will almost always perform better in the elution or removal of materials from a polar surface than either solvent alone. The combination of the solvents allows chemical selection of the lowest potential state for the system through local perturbation of the chemical composition. In most cases, the excess of the polar solvent in the presence of a contaminant will displace the contaminant from the surface, and the polarizable non-polar solvent will work to accommodate the contaminant into the liquid phase by a co-solvation resulting from the intermolecular interactions with the polarizability.

## **Cleaning options**

In the process of defining cleaning procedures for surfaces, it is required that the efficacy of the cleaning process, as well as its impact of the surface be evaluated. In no case should a cleaning process damage the article being cleaned, nor should the process leave residue. These are the key elements to designing a cleaning process. If these behaviors are validated then the part will be no worse coming out than when it went in. The worst-case scenario then would be finding an adequate cleaning process for the part.

It is absolutely mandatory that the vessels used for cleaning parts and optics is pristine. This means that the cleaning equipment including the clean room wipers and swabs be free of residual molecular and particulate material. Thus, prior to use, the equipment must be cleaned by the same or a more vigorous cleaning technique than the method being used to clean the parts. Typically if parts are to be cleaned using a wet technique that the parts are either washed with detergent or chemical cleaner or ashed using a high temperature furnace.

### **Protection of clean surfaces**

Clean surfaces must be protected before, during and after the cleaning procedures. Prior to cleaning there is the likelihood of surface damage due to scratch damage caused by hard particulates on the surface, and many optical coatings are subject to damage due to contaminants carried on skin. Thus, it is critical that when parts are handled, that they be handled wearing cleanroom garments. In addition, it is critical that the selection of gloves for use in handling be of a type that have minimal contact transport, and that there are no labile contaminants on the gloves. Further, plastic and rubber gloves are in many cases permeable to skin oil, and thus must be changed on a regular basis. Due to surface pick up of contaminants, it is also necessary to replace gloves due to the significant risk of contamination being carried by or transported through the gloves.

Gloves should not be allowed to be in contact with solvent due to the potential for extraction of material from the gloves, as well as degradation of the gloves, and

increasing the permeability of the gloves. The only generally acceptable glove for use with solvents is polyethylene. In this case, after applying the polyethylene gloves over gloved hands, the surface must be cleaned due to the limited particulate cleanliness of the gloves. A more difficult alternative that abates much of the particulate concerns is to reverse the polyethylene gloves so that the heat seams are inside the gloves, and the interior material will have less exposure to particulate contamination initially. But washing, wiping or rinsing of the polyethylene gloves is still recommended.

It is always best to make use of handling tools rather than gloved hands for handling parts. This prevents accidental face touching, or other contamination source due to touching something dirty and contaminating the gloves and then contaminating parts. Handling tools need be no more specialized than plastic tongs or forceps. The use of metal forceps or tongs is discouraged due to possible damage to parts, especially optics.

#### Pyrolytic Cleaning

Pyrolytic cleaning of borosilicate glass containers (beakers) is the best method for assuring that the containers are not contaminated with organic materials. Heating the containers from ambient temperature to a temperature adequate to combust all organic contaminants, after a quick detergent wash and three deionized water rinses leaves a contaminant free container. An exemplary scenario is: place the containers in the furnace on a stainless steel porous support with the opening down, slowly heat the containers (less than 10 degrees C per minute) to a final temperature of 550-600 degrees C, dwell there for two hours then cool down at about 3 degrees C per minute. This has been effective for removing grams of organic contamination, (it is not recommended for removing this gross of an amount of contamination due to emissions.) Quality assurance of the efficacy of this method is done by marking on the glass with a black permanent marker. When the beakers come out, the carbon black from the ink should be gone, and the beaker can be trusted to be clean. The notable exception is if the container had significant amounts of silicone contamination, in which case, a light dusting of fumed silica will be present in the beaker. If this occurs the beaker can be recleaned with detergent and solvent. The bigger issue at this point is the source of the silicone contamination.

#### Chemical Cleaning

Chemical cleaning of glassware is usually not recommended due to the highly corrosive nature of chemical cleaning systems. Chemical cleaning solutions such as alcoholic potassium hydroxide and sulfuric acid based cleaning solutions are best left for the chemical laboratory, and not for precision cleaning technicians. If the glassware is contaminated sufficiently to require chemical cleaning, throw it away or use the pyrolytic method above.

#### Detergent Cleaning

Detergent cleaning has great historical significance, and continues to hold great significance; the reason for this is that it works. Proper selection of detergent properties will allow the removal of both hydrophobic and hydrophilic materials from the surface of

an optic or other hard surface. The efficacy of the detergent is largely due to the combination of the polar head of the molecule and the non-polar or lipophilic end of the detergent. This allows the detergent to both solublize and disperse ionic or polar materials and non-polar materials. This gives the detergent the capability of performing many different actions.

The correct identification of materials properties and appropriate selection of the detergent is critical so that it does not affect the coatings. There is typically less risk in using basic laboratory detergents instead of ammoniacal acidic detergents. Ammoniacal detergents present the additional risks of hydrolysis of fluoride, sulfide, and selenide coatings and potential complexation reactions with metal oxides.

In the detergent cleaning of optical elements, the detergent should be prepared in deionized water and sub-micron filtered. The cleaning of optics should be carried out using a soft absorbent material to allow hard particulates to reposition themselves into the soft absorbent. In addition, the sorbent will pick up hydrophobic materials as well. After the part is suitably clean, the part should be rinsed in warm deionized water until the water draining from the part will no longer form bubbles, denoting the lack of residual surfactant. At this point the part can be blown dry with liquid nitrogen boil off through a deionizing blowgun.

#### Solvato-Chromic Cleaning

Solvato-Chromic cleaning is differentiated from solvent cleaning, as typical solvent cleaning rules are not directly applicable. Attempting to remove hydrocarbon vacuum pump oil from an optic using hexane will not be very effective, as the surface adsorption of the oil will not be readily broken using hexane. This is a result of the increase in polarizability with molecular mass. Thus, although like dissolves like in the bulk sense, the bulk material is unlike the surface adsorbed layers. Treating the contaminated surface as a chromatographic system is more applicable. In this view, the purpose is to displace the contaminants from the surface and provide adequate solubility for the contaminant in the liquid phase to prevent it from displacing the polar component of the solvent system from the surface. As another example, it is well known that perfluoroether lubricants are not soluble in solvents other than perfluorinated solvents or supercritical fluids. It is possible to displace high molecular weight perfluoroether lubricants from surfaces using chloroform and isopropyl alcohol 1:1 v:v. The perfluoroether is displaced from the surface and emulsifies. If not flushed off it will recover the surface, but it is displaced initially.

The second aspect of solvato-chromic cleaning is the adsorption or wicking of contaminants into a second high surface area material. In some cases, certain materials will resist rinsing as a method of removal with solvent. It is sometimes possible to utilize clean room wipers or other reasonably particle free high surface area materials to act as an alternative surface for adsorption. This is particularly effective with a mixed solvent system. In this case, the evaporation of solvent from the non-surface side will result in a net solvent flow into the wiper, resulting in a depletion of the contamination at the

surface. This is a very slow process and requires extremely clean wipers.

Flushing of the surface of a part with a solvent assuring that the part is rinsed from the top down is another effective cleaning method. In this case, multiple rinses of the part are required to assure the surface is clean. It should be noted that only fluorocarbon wash bottles should be used for cleaning. Polyethylene and polypropylene wash bottles contain extractable lower molecular weight polymer that will adhere strongly to nearly all surfaces. Testing of solvent extracts of fluorocarbon wash bottles has shown no more contamination than solvent blanks after weeks of extraction at room temperature. Extraction of the fluoropolymer bottles is recommended in any case as a verification step. Solvent remaining in wash bottles for more than one week should be discarded. It is even more important to never touch the tip of the wash bottle to anything. If this is done, rinse the tip and dispose of the solvent, then flush the tip several times then refill. Touching the tip of the wash bottle spout to a surface will result in contaminating the tip through contact and the solvent through back flow.

#### Thermal-Vacuum Cleaning

Thermal vacuum cleaning has been a mainstay of Aerospace contamination abatement for many years. This process involves placing the items in a high vacuum chamber and heating them. The purpose is to elevate the partial pressure of the contaminants in the system and then remove them through the use of a trap. In poorly designed and maintained systems, there is often the risk of contamination of the parts. In a well designed well maintained system, the risk of recontamination is far less than the risk of damage by any other means. In addition, in a well-instrumented chamber, it is possible to determine the point at which no redeposition of contaminants from the parts can occur.

A well-designed chamber will be constructed with an independently thermally controlled box oven. This will have a number of controlled area ports with a cold finger, scavenger plate, and one or more temperature controlled quartz crystal microbalances or surface acoustic wave devices. Each of these components should have independent thermal control, and the box oven should have thermal control as well as a thermal limit switch. Inside the box oven there should be baffles to allow equilibration of the internal distribution of the partial pressures to allow precise measurement of the outgassing, without the bias of a line of sight exposure.

The entire box oven should be encircled by a heatable cryogenic shroud, such that the internal box does not have a view to the external chamber walls that could result in transfer to the box oven. An external vacuum chamber can be heated for removal of surface contamination from the total system is a plus. Provision should be made for backfilling the chamber with cryogenic gas boil off through a sub micron filtered fill line. The door for the chamber should open into a class 1000 or better environment to maintain particulate cleanliness.

The pumping system should be ideally irreversible. Thus, there will be no chance of back streaming material into the chamber from the pumping system. There are a number of dry



pumping systems, capable of roughing out a chamber and meeting the requirements of the system, however, the use of pumping systems such as sorption pumps, cryo-pumps and ion pumps are attractive as they are inherently stronger pumps for contaminants, and they do not present the risk of oil based roughing pumps and diffusion pumps. Valving of the pumping system should provide two forms of feedback to the operator as to the position of the valve, and should automatically close upon loss of power, or other such system fault.

#### Purge Cleaning and Purged Storage

Purge cleaning is a close analog of thermal-vacuum processing. A purge system is an extremely simple to design build and operate. In addition, the purge systems can meet levels equal to or better than that attainable with thermal vacuum processing at a significantly lower total investment.

The basis of a purge system is a container, typically metal, that contains an inlet and an outlet and can be heated to accelerate the outgassing of materials. The behavior of the non-volatile residues in a purge system are identical to that seen in gas-solid chromatography, and can be so modeled, and can in fact be detected using the same technology, or captured using sorbent typically used for introduction of trapped contaminants from gas streams.

In the construction of a purge box system, all that is required is a clean flow control, a cryogenic liquid boil off gas source and an oven in which the parts will fit. This will allow the cleaning of parts to extremely clean levels. One simply flows gas through the box and allows the partial pressure reduction of the gas dilution to remove the contaminants over time. Additional hardware for this type of system to assure the security of the parts would be an over limit controller, check and relief valves, and potentially a sorbent trap for the incoming gas. It is extremely important to assure that all fittings and valves are disassembled and cleaned to remove the silicone used for lubrication of the packing and as a mold release during the manufacture of the protective caps, as well as processing oil and other contaminants from the tubing. These systems should usually be made using stainless steel, or other clean metal and any screw holes used to attach lids etc. should not penetrate into the interior of the box.

In these systems, in addition to the effect of the depletion of the contaminant partial pressure through dilution, there is also a limited exchange of the adsorbed contaminants for adsorption of the gas. This is known to show a small but measurable effect in adsorption chromatography.

Purged storage is probably the best option available for optical and optical hardware. Upon standing, optics and optical mounts and other parts will accumulate contaminant from other surfaces due to the surface energy of the clean surfaces. This can often occur due to outgassing of other parts, adhesives, packaging, even from the storage facility itself. By the identical reasoning used with purge cleaning, purge storage will remove contaminants from the stored materials, at a lesser rate. A positive side effect is that the

dry purge gas from the cryogenic gas boil off will dry out the optics thereby stabilizing the subsurface damage, and strain induced changes in the optics by eliminating the requisite water for the stress relaxation of the glass. On the downside, sol-gel coatings will need to be equilibrated to the working humidity before use, to allow the optics to meet their original performance requirements.

## **Analysis of Contamination**

There are numerous techniques that may be used for the analysis and determination of trace levels of NVR contamination. These include surface analytical techniques and techniques that require sampling, as mentioned above. Surface analytical testing although potentially more valuable, is also higher risk, due to the part having to be handled more, and subject to more environmental stress. Sampling increases the uncertainty of the other techniques, however, sampling will typically leave the sample surfaces pristine, as it must to quantify the contamination level.

Direct measurement techniques for optical and parts are widely varied. Techniques suitable for one component may not be suitable for another. For non-conductive and conductive parts, there are a few techniques suitable for analyzing the surfaces, these include ellipsometry, X-ray Photoelectron Spectroscopy, Photoelectron Spectroscopy and laser induced fluorescence. Restricting oneself to conductive parts, the use of Auger electron spectroscopy, or scanning electron microscopy with detection of electron induced x-rays results in no issues. In the absence of conductivity, sample charging resulting in signal loss, or damage to the sample of interest due to charge-induced breakdown can occur, at a level less than expected.

As mentioned above sampling of surfaces result in significantly less risk to the parts. However, there is more inherent uncertainty in the data due to the potential for incomplete sampling. In most cases, the sampling of surfaces will provide the best information in a reasonable time frame. This is in part due to the similarity of one sample to the next, and the potential for automating or batching a number of samples at one time.

Within the ultra trace regime, it is worthwhile to control the source of sample containers, and solvent supplies. For each set of samples run in one day, there should be at least one solvent blank, taken from the rinse solvent container, and of similar volume to the rinses. This allows direct comparison of the contaminant level in the solvent, and the level in the samples. Typically the solvent samples and rinses will be evaporated at slightly above room temperature in a chemical vapor hood. This is best done stepwise into smooth sided aluminum sample pans that have been pyrolytically cleaned and verified to the sensitivity available. A blank pan at ambient temperature should be follow the rest of the sample pans throughout processing of the rinse samples, this will account for adsorption of contaminants from the laboratory air. The mass of residue in each sample pan should be determined for quantitative use, sample dilution use and for identification of the analysis order.

In most analytical systems, there is the potential for carry over between samples. In many

cases, the individual rinse sample is irreplaceable, and must not be lost. Therefore, it is wise to run one or more blank samples and one or more known samples to assure that the equipment is operating properly prior to committing a sample. In the analysis of samples, the lowest concentration sample should be analyzed first. Thus, if there is carryover, it will be smaller than the contribution of the due to the next sample.

Following up

After all of the samples have been analyzed, the original contamination level of the part should be calculated, and the potential sources of the contamination identified. Armed with this information it may be possible to prevent catastrophe by correcting process, environmental or behavioral issues that result in additional contamination.

## **Summary**

The cleaning process development information enclosed provides a guide to the development of a cleanliness development and assurance program capable of attaining and maintaining contamination levels better than or equal to 50 A/10000, per the obsolete MIL-STD-1246C. This level of cleanliness is not possible to be attained by historical methods such as those mentioned by Bennett. Only through the methods developed within this laser project, and the knowledge of the surface behavior of the contamination, modeling of the contamination and the close interaction of all of the parties involved was this possible.

In addition, with a proper starting point interaction between the materials, mechanical, optical, laser, and contamination engineering staffs, there is great potential for improving the contamination levels attainable, or at least lowering the cost of attaining and maintaining the presented levels.

Measurement of the cleanliness of the cleaned surfaces is required. In this as in the other steps, there is risk associated with the process. The risk of damage to optics and other determination schemes is real and present. Likewise, there is risk of significant uncertainty in sampling of surfaces for contamination. It is mandatory that the surface chemistry of the system be well understood.

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